



# Intercomparison of far-infrared transmittance measurements

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**Abstract:** We present the results of the first systematic “round-robin” comparison of far-infrared transmittance spectra measurements, which was performed by five laboratories and piloted by Physikalisch-Technische (PTB). The transmittance spectra of four different samples were measured by the participating laboratories in the 600 cm<sup>-1</sup> to 10 cm<sup>-1</sup> range (16.67 μm to 1000 μm) in a blind comparison. Different types of instruments, Fourier transform infrared (FT-IR) spectrometers of Michelson type and a laser radiation-based system were used for the transmittance measurements. FT-IR spectrometers are the most popular and commonly used instruments for the spectral characterization of materials in the infrared spectral range, and are well established for quantitative measurements in the mid- and near-infrared spectral ranges. However, obtaining quantitative transmittance measurements in the far-infrared spectral range by means of these instruments is challenging, because it involves weaker radiation sources, stronger diffraction effects, significant radiation originating from the sample itself and temperature gradients inside the spectrometer that may not be given proper consideration. Therefore, this comparison was initiated to test the actual capability of and identify problems with FT-IR transmittance measurements in this spectral region. We discuss the results and the possible reasons for the observed discrepancies.

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## 1. Introduction

Due to their superior properties compared to dispersive instruments (i.e., their high throughput and fast and wide spectral coverage), Fourier transform infrared (FT-IR) spectrometers have become the most commonly used instruments for the spectral characterization of materials in the entire infrared spectral range. Commercially available instruments are able to achieve very good stability and signal-to-noise ratios.

However, the use of FT-IR spectrometers for quantitative radiometric applications is complicated; possible pitfalls include the non-linearity of the detector, multiple reflections in the interferometer block and the thermal emission of all instrument components. These effects are more dominant in the far-infrared (FIR) spectral range than in the near- and mid-infrared spectral ranges; when taken together with increasing diffraction and weaker radiation sources, these effects make the quantitative measurement of optical properties of materials challenging in the FIR spectral range.

The optical properties transmittance and reflectance of a sample are defined as the flux of radiation transmitted through or reflected from this sample with respect to the incident flux. Despite these simple definitions, the actual measurement of FIR optical properties is not without difficulty due to the effects listed above arising in FT-IR spectrometers.

To test the actual quality of transmittance measurements in the FIR spectral range, five partner laboratories, Physikalisch-Technische Bundesanstalt (PTB), Max-Planck-Institut für Radioastronomie (MPIfR), Institut für Photonische Technologien e.V. (IPHT), Deutsches Zentrum für Luft- und Raumfahrt (DLR) and Optotransmitter Umweltschutz Technologie (OUT), initiated a blind round-robin comparison. Below, an overview of the investigated samples and applied instrumentation is presented, and the resulting transmittance spectra of all participants are compared for each sample.

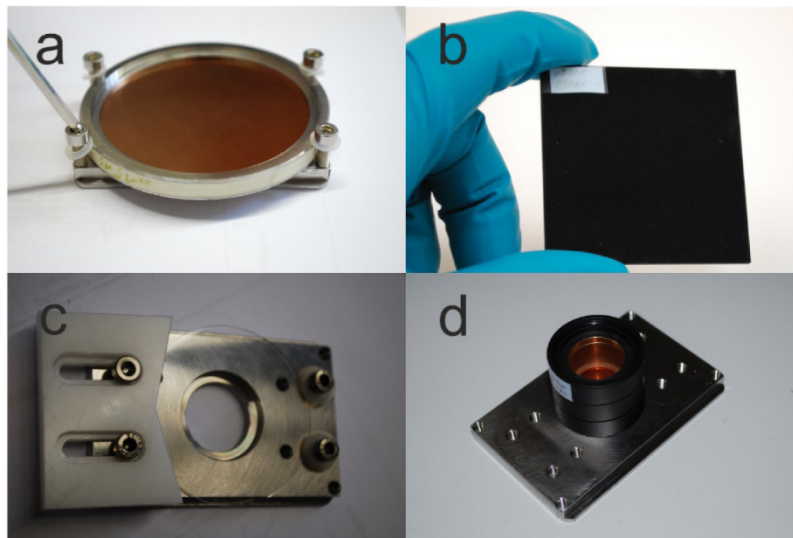


Fig. 1. Pictures of the investigated samples, no. a, c and d show the sample mounted in the holder used within the comparison: a: LP 100, b: NG1, c: Quartz Disc B and 4: High Pass 3.

## 2. Experimental

### 2.1 Samples

The participants selected the samples according to four requirements: the samples should have pronounced spectral features in the FIR spectral range; they should be commonly used in the FIR spectral range; they should be easy to handle with respect to the spectrometer dimensions; and they should influence the beam shape as little as possible.

Four samples were chosen: two low-pass filters (lower wavenumbers are transmitted) whose cut-on edges were at approx.  $250\text{ cm}^{-1}$  (Quartz Disc B) and  $100\text{ cm}^{-1}$  (LP 100); one volume absorber that starts to become transparent at wavenumbers below  $25\text{ cm}^{-1}$  (NG1) and that, in principle, could also be considered a low-pass filter; and one high-pass filter (higher wavenumbers are transmitted) whose cut-off edge was at about  $136\text{ cm}^{-1}$  (High Pass 3). The mounted samples are depicted in Fig. 1. Below, a brief description of the filters and the absorber is given.

#### LP 100

The LP 100 low-pass filter is a commercially available product made by QMC Instruments [1]. This filter consists of a stack of metal meshes and dielectric foils mounted in a metal ring, as shown in Fig. 1(a). Since the publication of Ulrich [2] on the transmittance properties of metal grids, metal mesh filters have been commonly used in the FIR spectral range.

The thickness of  $(225 \pm 5) \mu\text{m}$  of this sample is mainly due to the dielectric substrate, making it a relatively thin sample. Consequently, its influences on the beam shape and beam path, as well as a possible magnification of the spot on the detector, should be negligible.

### NG1

NG1, a neutral-density filter glass made by SCHOTT AG and shown in Fig. 1(b), is typically used in the FIR spectral range as a volume absorber. Due to the good uniformity, the optical quality and the polishing of the surfaces of the NG1 material, scattering can be neglected. Nearly the complete spectral range of the penetrating radiation from visible to FIR is absorbed by the NG1 material. However, at wavenumbers below  $25 \text{ cm}^{-1}$ , NG1 becomes transparent. The optical properties of NG1 are well known in the visible, near- and mid-infrared spectral ranges up to  $5.2 \mu\text{m}$  (approx.  $1800 \text{ cm}^{-1}$ ) according to the data published by its manufacturer. Above this wavelength, no manufacturer data is available. However, the NG1 transmittance in the FIR spectral range was measured in previous work [3] using a vacuum FT-IR spectrometer – namely, a VERTEX80v made by the Bruker Corporation. The thickness of the sample was  $(1.087 \pm 0.004) \text{ mm}$ .

Due to the high absorbance of the sample, especially at higher wavenumbers, it was expected that its transmittance measurement would show the largest difference between the reference measurements and the measurements with the applied sample under test. Therefore, this sample was a critical indicator for deviations in the comparison resulting from the different dynamic ranges and nonlinearities of the applied detectors.

### Quartz Disc B

Quartz Disc B is presented in Fig. 1(c). The thickness of this quartz disc is  $(1.003 \pm 0.003) \text{ mm}$ . The disc has the largest optical thickness of all the samples used in this comparison. Because of its transparency, multiple reflections inside the sample occur, leading to a strong spectral modulation of the transmitted radiation. For this reason, a reliable mounting procedure was essential in order to obtain comparable results. In particular, it was necessary to ensure that the same angle of incidence was realized by all participants. To reduce the back reflections from the interferometer block, which were expected to have a critical influence for this sample in particular, a slightly slanted mounting angle of about  $1.5^\circ$  was chosen. Possible deviations in the comparison were expected from different solid angles of the radiation incident on the sample within the different applied instruments, as well as from a slightly defocused spot on the detector due to the difference in optical path length between the reference and sample measurements.

### High Pass 3

High Pass 3 is a high-pass filter in the form of a freestanding metal mesh. It consists of densely packed hexagonal holes in a copper foil that resemble a honeycomb. The grid constant of the mesh is  $48 \mu\text{m}$ , the wall width is  $8 \mu\text{m}$  and the thickness of the sample is  $(111 \pm 7) \mu\text{m}$ . In spite of their delicate structure, honeycomb meshes are mechanically very robust. Structures of this type have been developed as FIR filters for the ISOPHOT project, and were fabricated with the help of the LIGA process [4]. In this way, the walls of the single unit cell show an extremely high aspect ratio and the entire array is very uniform; at the same time, the mesh has plane surfaces of unique evenness.

The array of hexagonal holes is a special array form of waveguides. From this point of view, it can be easily understood that the filter blocks frequencies below the cut-on of the single waveguide and becomes transparent at higher frequencies. The hexagonal hole resembles a cylindrical hole. Arrays of cylindrical holes – known as *thick grill filters* – were intensively investigated in [5]; their optical properties were calculated from geometric parameters in [6]. Theoretically, the transmittance of this grill filter should be on the order of  $10^{-8}$  for very low frequencies. The grill filter starts to become transparent for the TE<sub>11</sub> mode,

which is close to  $136\text{ cm}^{-1}$ , and then becomes maximally transparent before diffraction effects set in at higher frequencies reducing the transmittance again. Thus, the sample offers three regions of transmittance: a region with ultimate blocking whose transmittance is on the order of  $10^{-8}$ ; a high-frequency region whose transmittance is close to unity; and, in between these regions, a very steep transition region around  $136\text{ cm}^{-1}$ .

For the blocking region, it was expected that spectrometers – even if equipped with the most sensitive detectors in this spectral region, such as helium-cooled bolometers – would not be able to resolve the low transmittance value. Thus, the measurement here merely gives an impression of the lowest radiometric limit of the system. In the transition region – mainly for the TE11 mode – it was expected that the spectral resolution (lowest intended spectral resolution  $0.4\text{ cm}^{-1}$ ) would not be sufficient, possibly resulting in smoothed details of the spectral structure of the filter transmittance.

This filter was mounted in a tube (seen in Fig. 1(d)) and was measured under normal incidence. Difficulties with this sample may arise from back reflections in the different instruments.

## 2.2 Comparison procedure – round-robin

First, the four samples chosen were measured by the pilot laboratory (PTB). Then, PTB sent the samples, along with their sample holders, an accessories kit and a detailed mounting description, to the other participants in the intercomparison. The order of the measurements performed in this round-robin comparison was as follows: PTB; MPIfR; IPHT; OUT; and DLR. Finally, PTB re measured the samples in to detect possible aging effects. The following measurement conditions were agreed on between the partners: The spectral wavenumber (wavelength) range of interest was partitioned into two ranges: range I :  $600\text{ cm}^{-1}$  to  $50\text{ cm}^{-1}$  ( $16.67\text{ }\mu\text{m}$  to  $200\text{ }\mu\text{m}$ ), measured with a resolution of  $2\text{ cm}^{-1}$ ; and range II:  $50\text{ cm}^{-1}$  to  $10\text{ cm}^{-1}$  ( $200\text{ }\mu\text{m}$  to  $1000\text{ }\mu\text{m}$ ), measured with a resolution of  $0.4\text{ cm}^{-1}$ . Each FT-IR measurement, with an average of 256 scans, was repeated 3 times. Additionally, the following internal parameters for the FT-IR spectrometers were predetermined: Blackman-Harris 3 Term apodisation; Mertz phase correction; and two-fold zero-filling. A set of possible beam splitter/detector combinations was proposed.

With these measurement conditions defined, it was assumed that the measurement period for each partnering laboratory would be around two weeks. After the measurements, each participant sent the results to the pilot laboratory only, which was responsible for the evaluation of the data.

## 2.3 Experimental setups

Three different models of spectrometers were used to perform the transmittance measurements throughout the comparison: Commercial FT-IR spectrometers of the VERTEX80v and VERTEX70v types made by the Bruker Corporation, as well as a DA3.26 made by ABB-Bomem. Furthermore, transmittance measurements were performed with a THz laser at selected laser emission lines.

VERTEX70v and VERTEX80v FT-IR spectrometer features an evacuated optics bench in order to avoid interferences due to atmospheric absorptions and in order to achieve greater stability. They can be operated in a high resolution mode, a fast rapid scan mode and a step scan mode. The instruments can optionally be equipped with different optical components to cover the spectral range from ultraviolet to FIR. In standard configuration, the VERTEX80v and in its special version the VERTEX70v provide a resolution of better than  $0.2\text{ cm}^{-1}$ .

Below, the measurement instrumentation of all participants is given in the order of their participation in the round-robin comparison.

### 2.3.1 PTB FT-IR measurements

The FT-IR measurements were performed using two different measurement setups of PTB. At the facility for spectral emissivity measurements, which is equipped with a VERTEX80v, the range from  $600\text{ cm}^{-1}$  to  $30\text{ cm}^{-1}$  was covered [7]. At this facility, a mercury-vapor lamp, two beam splitters (Mylar with a thickness of  $6\text{ }\mu\text{m}$  and  $50\text{ }\mu\text{m}$ , respectively) and two detectors (a room-temperature pyroelectric detector and a He-cooled Si-bolometer) were used. The second measurement setup, located at the Metrology Light Source (MLS) electron storage ring of PTB [8], also used a VERTEX80v. These measurements, which covered the spectral range from  $30\text{ cm}^{-1}$  to  $10\text{ cm}^{-1}$ , were also performed using a mercury-vapor lamp, but with two different beam splitters (Mylar with thicknesses of  $23\text{ }\mu\text{m}$  and  $125\text{ }\mu\text{m}$ ) and a pumped, He-cooled Si-bolometer.

Typical measurement approaches for optical transmittance and reflectance follow the definitions of these quantities and determine the quotient of the reflected or transmitted flux with respect to the incident flux. However, in the mid- and far-infrared ranges, this approach can easily lead to incorrect results if offsets resulting from background radiation, from the sample itself and from temperature gradients inside the spectrometer are not correctly considered. The observed deviations are particularly significant when working with cooled detectors and at low flux levels. PTB applied a measurement and evaluation procedure to correct these offsets. By measuring at two flux levels of incident radiation and calculating the transmittance as quotient of differences of the signals measured at the two flux levels, and by carefully temperature stabilizing the spectrometer, the offsets can be canceled. In this way, the transmittance and reflectance can be determined in the range from  $400\text{ cm}^{-1}$  to  $10\text{ cm}^{-1}$  with reduced systematic errors [9]. Details of this approach and examples are given in [9]. However, compared to [9] the flux levels used in this study were significantly higher resulting in smaller corrections.

### 2.3.2 PTB laser measurements

The transmittance of the samples was measured by means of laser radiation at the THz detector calibration facility of PTB, which is described in [4]. The applied laser system can provide radiation at several frequency settings in the range from 1 THz to 5 THz, five of which were used in this case: 1.04 THz ( $34.7\text{ cm}^{-1}$ ), 1.4 THz ( $46.7\text{ cm}^{-1}$ ), 2.52 THz ( $84.0\text{ cm}^{-1}$ ), 4.25 THz ( $141.8\text{ cm}^{-1}$ ) and 5.67 THz ( $189.1\text{ cm}^{-1}$ ). The applied THz detector, a special thermopile detector, is described in detail in [3]. The measurement at each frequency setting was performed according to the quality management-approved rules and regulations specified in ISO17025. The relative standard uncertainty of the calibration at these frequencies is 2% (expansion factor  $k = 1$ ) and takes the spatial uniformity of the detector into account.

### 2.3.3 MPIfR

The Bomem spectrometer operated at MPIfR is dedicated to measurements in the very long wavelength regime (mm waves and sub-mm waves); its best standard resolution is  $0.02\text{ cm}^{-1}$  (apodized). It is a rapid scanner and has an evacuated optical bench. The source of the spectral region covered is an HPK125 mercury-vapor lamp that is kept at a constant temperature ( $\pm 0.1\text{ K}$ ) by means of water cooling.

For the spectral regions under consideration, three different measurement setups were used: in Range I ( $600\text{ cm}^{-1} - 30\text{ cm}^{-1}$ ) a DTGS detector plus a (standard) preamplifier combined with a Ge-coated  $6\text{ }\mu\text{m}$ -thick Mylar beam splitter; in the first part of Range II ( $50\text{ cm}^{-1} - 15\text{ cm}^{-1}$ ), a DTGS detector plus longpass filters plus a modified preamplifier combined with a  $50\text{ }\mu\text{m}$ -thick Mylar beam splitter; in the second part of Range II ( $25\text{ cm}^{-1} - 3\text{ cm}^{-1}$ ), a He-cooled Ge bolometer ( $T \approx 1.3\text{ K}$ ) combined with a  $100\text{ }\mu\text{m}$ -thick Mylar beam splitter.



The filters, the preamplifier and the Ge bolometer were developed at MPIfR.

#### 2.3.4 IPHT

IPHT performed the measurement using the Bruker VERTEX80v vacuum spectrometer. The Fourier spectrometer at IPHT can measure in a wavelength range between 0.4  $\mu\text{m}$  and 2 mm ( $25000\text{ cm}^{-1}$  to  $5\text{ cm}^{-1}$ ). The measurements were performed using two different setups. For the range from  $600\text{ cm}^{-1}$  to  $50\text{ cm}^{-1}$ , a globar, a beam splitter with a thickness of 6  $\mu\text{m}$  (Mylar Multilayer) and a room-temperature pyroelectric detector (DTGS w/PE) were used; for the measurements of the second region from  $50\text{ cm}^{-1}$  to  $10\text{ cm}^{-1}$ , a mercury-vapor lamp, a beam splitter with a thickness of 50  $\mu\text{m}$  (Mylar 50 Micron) and a He-cooled Si bolometer including a  $\text{NEP} < 10^{-12}\text{ W Hz}^{-0.5}$  were used.

#### 2.3.5 OUT

All FT-IR measurements were performed using a VERTEX70v made by Bruker Optics that uses a so-called Rocksolid instead of a classical Michelson interferometer. Here, in contrast to the use of flat mirrors, a Rocksolid incorporates two retroreflecting corner cube mirrors in an inverted double pendulum arrangement, thus desensitizing it to tilting but limiting the spectral range to  $28000\text{ cm}^{-1}$ . The VERTEX70v used at OUT e.V. is a special version that is equipped with a high-resolution feed (improved resolution of  $0.16\text{ cm}^{-1}$ ) and that utilizes only Al mirrors.

The investigations were performed by means of two measuring setups.

In Range I ( $600\text{ cm}^{-1}$  to  $50\text{ cm}^{-1}$ ), with a globar (or, alternatively, a mercury vapor lamp) as the radiation source, beam splitters (Mylar with a thickness of 6  $\mu\text{m}$  or, alternatively, a Si beam splitter) and a room-temperature pyroelectric DTGS detector were used. The measurements in Range II between  $50\text{ cm}^{-1}$  and  $10\text{ cm}^{-1}$  were carried out with a mercury vapor lamp, a Si beam splitter and a He-cooled Si bolometer. All measurements were performed under vacuum and at room temperature. For sample holding, an A 480 direct transmission unit made by Bruker Optics was used, ensuring that parallel light passed the sample. In order to ensure reproducible measuring conditions, a lifting device developed by OUT was used in combination with a special feedthrough in the lid of the sample chamber. This device enables the removal of the samples from the beam path for background measurements and their repositioning for the sample measurement without breaking the vacuum.

#### 2.3.6 DLR

The FT-IR measurements at DLR were performed by means of a VERTEX80v spectrometer. Over both spectral ranges (i.e., the range from  $600\text{ cm}^{-1}$  to  $50\text{ cm}^{-1}$  and the range from  $50\text{ cm}^{-1}$  to  $10\text{ cm}^{-1}$ ), a mercury-vapor lamp served as the blackbody source. In the long-wavelength range, a Mylar beam splitter with a thickness of 50  $\mu\text{m}$  was used. In this range, a sensitive He-cooled Si bolometer (Infrared Laboratories) was used as the detector. Due to a very small detector signal in this spectral region, the aperture in the intermediate focus was set to the largest possible value (8 mm). To reduce the thermal load of the detector and to avoid interference of unwanted radiation from shorter wavelengths, a low-pass filter (cut-off wavenumber:  $100\text{ cm}^{-1}$ ) was mounted in front of the cryostat. In the range from  $600\text{ cm}^{-1}$  to  $50\text{ cm}^{-1}$ , a DTGS detector at room temperature yielded a sufficient signal-to-noise ratio. The broadband beam splitter in this region was a typical, coated 6  $\mu\text{m}$  Mylar foil. The aperture in the intermediate focus was also set to the largest possible value (8 mm).

### 3. Results and discussion

The results of the comparison are visualized in a series of figures for each sample as follows: First, for both spectral ranges, an overview of the results of all participants is shown. For

better comparability, all of these figures have the same y-(transmittance) scale, and all Range I and Range II measurements feature a common respective x-(wavenumber) scale. For a more detailed examination of the data, two composite figures are then provided for each sample and each spectral range that show the arithmetic mean of all measurements in the upper part and the deviation of the data of each participant from the arithmetic mean in the lower part. Data which showed a significant deviation from the majority of the measurements was not used in the calculation of the mean and is discussed individually.

### 3.1 LP 100

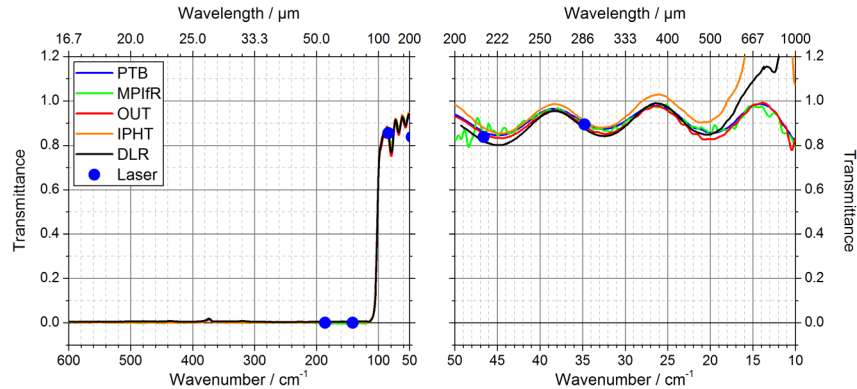


Fig. 2. Measured spectral transmittances of all participants of the low-pass filter LP 100 in Spectral Range I (left) and Spectral Range II (right). Blue dots indicate THz laser measurement results.

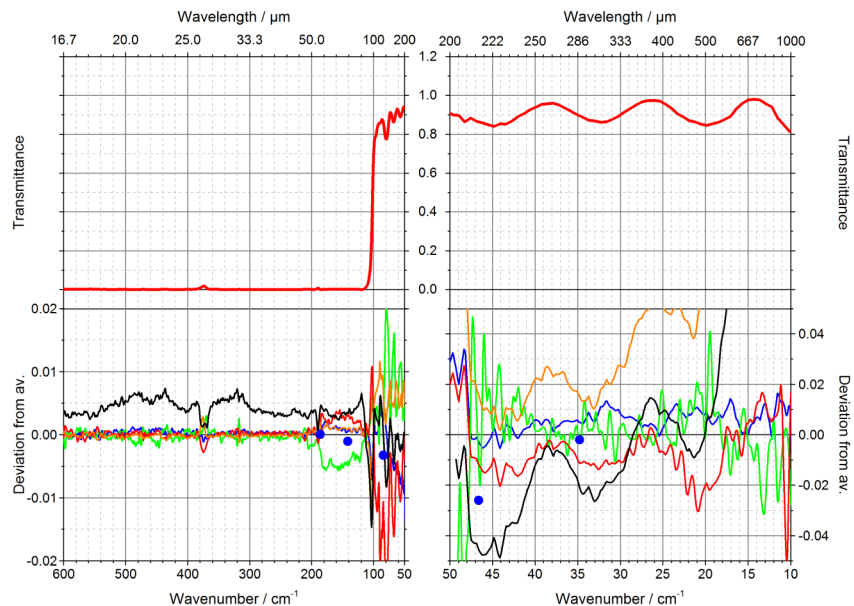


Fig. 3. Measured spectral transmittances of low-pass filter LP 100 in Spectral Range I (left) and Spectral Range II (right). Top: arithmetic mean of the participants. Bottom: deviation of the individual spectra from the arithmetic mean. Colors as in Fig. 2.

The transmittance of the LP 100 filter measured by all participants in the range from  $600\text{ cm}^{-1}$  to  $50\text{ cm}^{-1}$  is given on the left-hand side of Fig. 2. The bottom left-hand section of Fig. 3 shows a very good agreement of the data in the higher wavenumber range, which is

dominated by a broad blocking area down to approx.  $110\text{ cm}^{-1}$ . The filter becomes transparent for smaller wavenumbers and has a transmittance of approx. 0.9 below  $100\text{ cm}^{-1}$ . In the blocking area, the transmittance measured by DLR (black) is significantly larger than the transmittance measured by all other participants (Fig. 3). According to DLR, this deviation was due to the higher noise level of their DTGS detector. Therefore, the DLR results were excluded from the mean value, which is shown at the top of Fig. 3. In the transparent spectral region of the filter, the largest deviations from the mean value are about 0.02. The laser measurements (blue dots) are in very good agreement with the results of the FT-IR spectroscopy data.

The results in the lower wavenumber region shown on the right-hand side of Figs. 2 and 3 are slightly more spread. The filter has a high transmittance over the whole spectral range that is slightly spectrally modulated. The bottom right-hand side of Fig. 3 shows a good agreement of the data of all participants from  $50\text{ cm}^{-1}$  to approx.  $20\text{ cm}^{-1}$ . At about  $20\text{ cm}^{-1}$ , the transmittance results from IPHT (orange) and DLR start to rise above the value of 1, due to this large deviation; these results were therefore excluded from the mean value. For some measurements with the bolometer, DLR reported a signal saturation of the detector when one of the internal detector filters was used. Furthermore, the internal aperture of the spectrometer was set by DLR and IPHT to a larger value than those used by the other participants (IPHT: 12 mm; DLR: 8 mm, PTB: 2 mm; OUT: 4 mm). Because similar sources and detectors with similar responsivities were used among the instruments in the comparison, this may have led to smaller dynamic ranges compared to the other instruments. Thus, both experimental conditions could explain why the value of the transmittance was too high.

In summary, the results of MPIfR (green), OUT (red) and PTB (blue), and the results of the PTB laser (blue dots), deviate by less than about 0.04 from the averaged transmittance over the whole spectral range.

### 3.2 NG1

The left-hand side of Fig. 4 shows a very good agreement of the NG1 filter transmittance data in the higher wavenumber range. In practice, the filter can be considered to completely block the radiation in this range. A more detailed view given by the deviation from the mean plot in the lower left-hand section of Fig. 5 reveals a significantly higher transmittance measured by DLR in this range. Consequently, the DLR measurements were excluded from the mean value in this range. Here, the deviations are again explained by DLR as being due to a higher noise level of their DTGS detector.

The results in the lower wavenumber region show a good agreement of the measurements (right-hand side of Fig. 4). The transmittance of the NG1 filter rises for wavenumbers lower than  $25\text{ cm}^{-1}$ , and the mean value reaches a transmittance of approx. 0.4 at  $10\text{ cm}^{-1}$  (Fig. 5, right-hand side). Similarly to the LP 100 filter, the measured transmittance from IPHT deviates significantly below  $20\text{ cm}^{-1}$  to values above the mean. One possible explanation of the observed difference is that a saturation of the detector could be caused by the internal aperture of the spectrometer mentioned above, which was too wide. The spectrum of IPHT was excluded from the mean value because of this result.

The measured transmittances of MPIfR, OUT, DLR and PTB, and of the PTB laser, have a maximum deviation of about 0.05 from the averaged transmittance at  $10\text{ cm}^{-1}$ . Due to the large differences between sample signal and reference signal for this filter, we attribute these relative deviations of up to 25% to nonlinearities of the detectors.



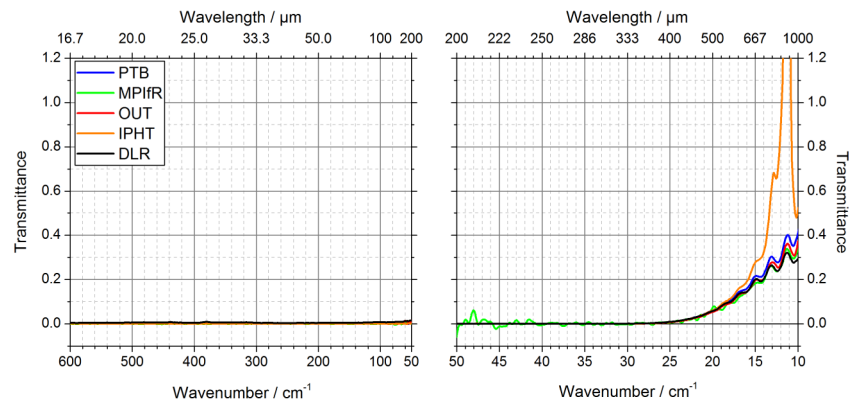


Fig. 4. Measured spectral transmittances of all participants of the NG1 volume absorber in Spectral Range I (left) and Spectral Range II (right).

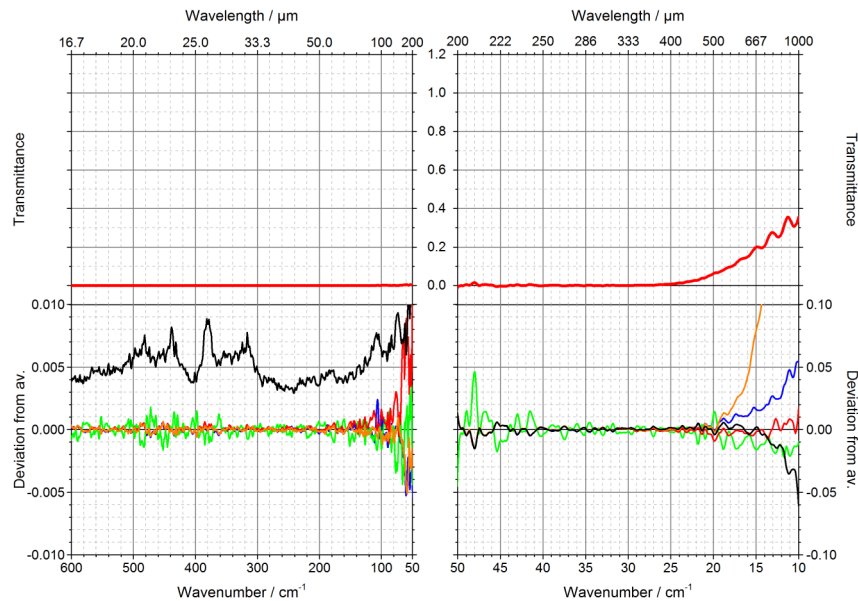


Fig. 5. Measured spectral transmittances of volume absorber NG1 in Spectral Range I (left) and Spectral Range II (right). Top: arithmetic mean of the participants. Bottom: deviation of the individual spectra from the arithmetic mean. Colors as in Fig. 4.

### 3.3 Quartz Disc B

Figure 6 shows a good agreement of all participants for most of the data of the Quartz Disc B filter in the higher wavenumber range; this data is split into a blocking area down to approx.  $250\text{ cm}^{-1}$  and an area with slightly rising transmittance from  $250\text{ cm}^{-1}$  to  $50\text{ cm}^{-1}$ , reaching a transmittance of 0.8 at  $50\text{ cm}^{-1}$  (see Fig. 7, left-hand side). In the blocking area, the difference from the mean value is less than 0.01 for all participants except DLR. Again, this deviation is explained by DLR as being due to noise level of their DTGS detector. In the transparent area, the laser measurements are remarkably higher than the average value for two wavenumbers. This could be explained by the much smaller band width of the laser measurements. Additional FTIR measurements with a higher resolution of  $0.1\text{ cm}^{-1}$  show a good agreement of the laser and the FTIR spectrometer results (gray in Fig. 6, left-hand side, and Fig. 7, top left-hand side). Below wavenumbers of about  $70\text{ cm}^{-1}$ , the result from OUT deviates much more than the results of all other participants, and was excluded from the mean value.

The results in the low wavenumber region show a fairly good agreement of the measurements (Fig. 7). The transmittance of the low-pass filter is high in the entire spectral range depicted, with a modulation between 0.6 and 1 (Fig. 7, top right-hand side). The result from IPHT deviates strongly below 20  $\text{cm}^{-1}$ . It seems reasonable to explain the difference as being due to the saturation of the detector caused by the internal aperture of the spectrometer, which was too wide.

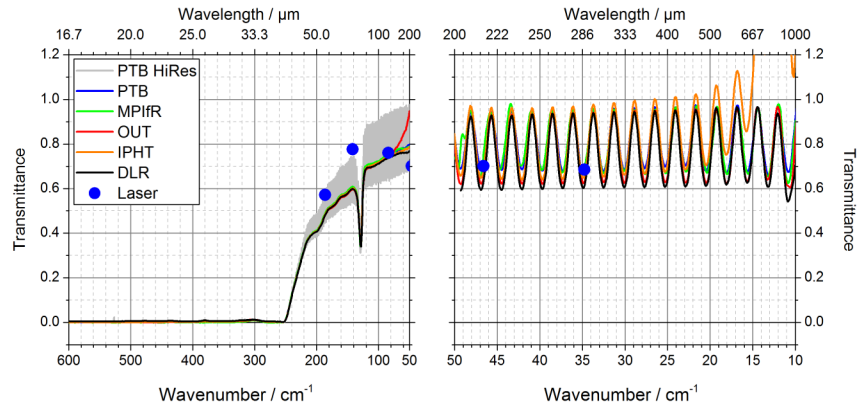


Fig. 6. Measured spectral transmittances of all participants of Quartz Disc B in Spectral Range I (left) and Spectral Range II (right). Blue dots indicate THz laser measurement results. Gray area on the left indicates spectral results with a resolution of 0.1  $\text{cm}^{-1}$ .

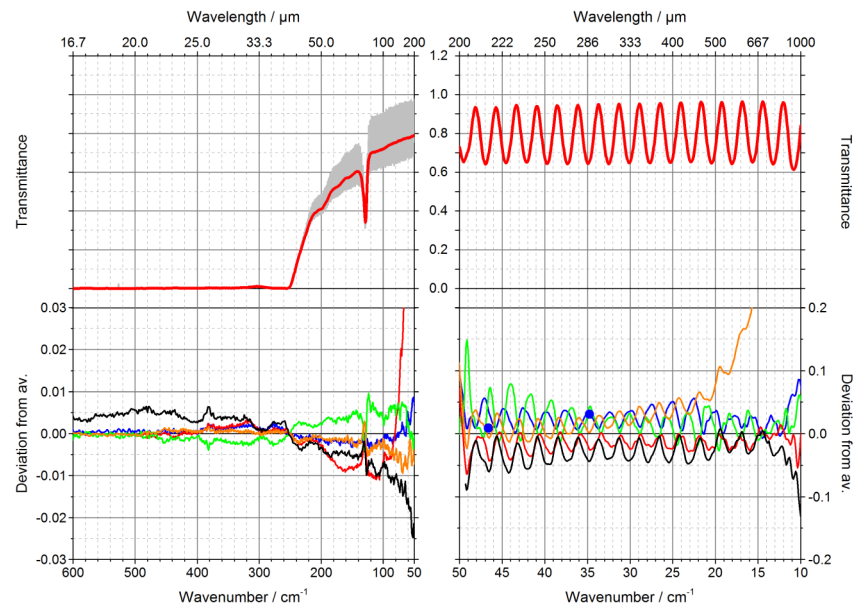


Fig. 7. Measured spectral transmittances of Quartz Disc B in Spectral Range I (left) and Spectral Range II (right). Top: arithmetic mean of the participants. Bottom: deviation of the individual spectra from the arithmetic mean. Gray area on the top left indicates spectral results with a resolution of 0.1  $\text{cm}^{-1}$ . Colors as in Fig. 6.

The results of MPIfR, OUT, DLR and PTB, and of the PTB laser, have a deviation of less than 0.1 from the averaged transmittance over the whole spectral range in Range II. The modulated deviations between the participants indicate a spectral shift of the modulated transmittance, which may result from an insufficient reproducibility of the sample mounting at the different instruments. In addition, different solid angles of the radiation incident on the

sample in the different types of spectrometers can also result in a difference of modulation depth.

### 3.4 High Pass 3

The measured transmittances of the High Pass 3 filter show the largest differences between the participants in this comparison. Figure 8 shows a good agreement of MPIfR, OUT and PTB in the high wavenumber range, which is dominated by a transparent region down to  $135\text{ cm}^{-1}$ , reaching a transmittance of 0.9 at about  $200\text{ cm}^{-1}$  (see Fig. 8, left-hand side). In the transparent region, the difference is less than 0.05 between these three participants. They also agree well with the results of the laser measurements. The results from IPHT and DLR deviated significantly from those of this group (up to 0.2 below), but also deviated up to 0.1 from each other. In particular, the large deviations at the steep transmittance drop at  $136\text{ cm}^{-1}$  indicate a slight spectral shift which might be caused by an angle displacement in the mounting of the sample. At this position also the deviation from one measurement obtained with the laser is very large indicating a steep slope and a probably a higher transmittance value which is smoothed to a lower value by the limited resolution of the FTS measurements.

In the blocking region, all participants agree better than 0.01 in most of Range II, as seen on the right-hand side of Fig. 9. DLR did not provide data for this range due to the high noise level they had in these measurements. Between  $45\text{ cm}^{-1}$  and  $50\text{ cm}^{-1}$ , the measurement of MPIfR shows a slightly alternating transmittance that was not observed by the other participants, who reported transmittances of  $10^{-3}$  or lower. Below  $15\text{ cm}^{-1}$ , the transmittances measured by MPIfR and PTB are  $10^{-4}$  to  $10^{-3}$ , which is remarkably good when compared to the predictions of the filter theory, while the measurements of OUT and IPHT deviate to larger values.

## 4. Summary

Laboratories of five institutions with experience in FIR spectroscopy took part in a blind round-robin comparison of spectral transmittance in the spectral range from  $600\text{ cm}^{-1}$  to  $10\text{ cm}^{-1}$  ( $16.67\text{ }\mu\text{m}$  to  $1000\text{ }\mu\text{m}$ ). The comparison was carried out using FT-IR spectrometers and a THz laser measuring the transmittance of four different samples, with PTB as the pilot laboratory.

It became evident that measurements in the FIR spectral range are still challenging, even when state-of-the-art, commercially available instrumentation is used. Below  $20\text{ cm}^{-1}$  in particular, deviations rise to 0.1 or 25%. Observed deviations were attributed to nonlinearities of the systems, alignment errors and to wide apertures. Some significant deviations were also found in the higher wavenumber range. These were also attributed to sample alignment and detector saturation. As described in the experimental section the participants use different evaluation procedures. This was intended because the comparison should reflect the situation how results compare, when participants use their established evaluation scheme.

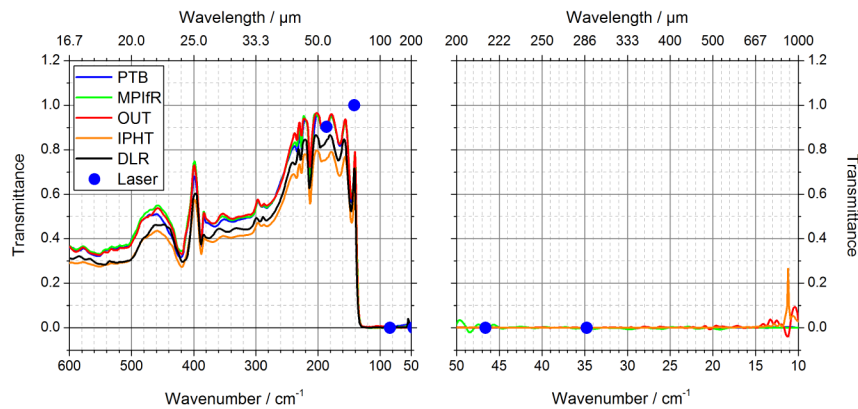


Fig. 8. Measured spectral transmittances of all participants of filter High Pass 3 in Spectral Range I (left) and Spectral Range II (right). Blue dots indicate THz laser measurement results.

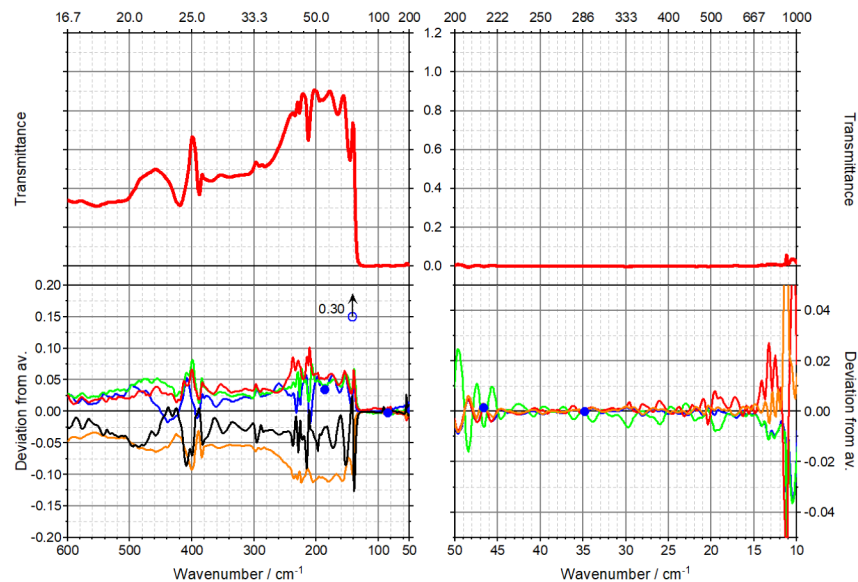


Fig. 9. Measured spectral transmittances of filter High Pass 3 in Spectral Range I (left) and Spectral Range II (right). Top: arithmetic mean of the participants. Bottom: deviation of the individual spectra from the arithmetic mean. Colors as in Fig. 8. One laser measurement is of the diagram and labelled accordingly.

In conclusion, the comparison showed considerable consistency of the measurement results in this spectral region, which is known for complicating effects such as diffraction and thermal radiation of the optical setup. The overall agreement in the transmittance spectra was remarkably good. In most cases, the agreement is better than 0.05, sometimes even 0.001. This shows the potential of the commercially available FT-IR spectrometers in combination with different optical elements and detectors. Furthermore, the spectra agree well with laser measurements as an independent technique indicating an absence of dominant systematic errors. To avoid systematic errors and improve the comparability of transmittance measurements in this spectral range the availability of appropriate reference samples would be desirable.

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